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The First Example of an Organogallium Compound Containing a Ga-Te Bond: Synthesis, Properties and Molecular Structure of $[(Me_3CCH_2)_2GaTePh]_2$

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Michael A. Banks, O. T. Beachley, Jr., Henry J. Gysling, and Henry R. Luss

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and
The Corporate Research Laboratories and
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Molecular Structure of $[(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}]_2$

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and Henry R. Luss^{1c}

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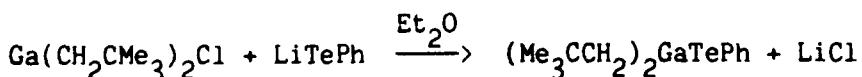
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Abstract

The first example of an organogallium compound with a Ga-Te bond $[(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}]_2$, which is stable at room temperature, has been synthesized and characterized by elemental analyses, IR, ^1H NMR and ^{125}Te NMR spectroscopy and an X-ray structural study. The dimeric molecule crystallizes in the monoclinic space group $P2_1/c$ with unit cell dimensions of $a = 18.864(7)\text{\AA}$, $b = 10.041(5)$, $c = 19.869(9)$, $\beta = 101.80(3)^\circ$, $V = 3684(5)\text{\AA}^3$, $Z=4$, $D_{\text{calcd}} = 1.505\text{g cm}^{-3}$, $R = 0.030$, $R_w = 0.035$. The four membered Ga_2Te_2 ring has a butterfly arrangement with the phenyl groups in the anti-position relative to the ring. The Ga-Te bond distances are in the range $2.7435(8)$ - $2.7623(8)\text{\AA}$.

Organometallic compounds of gallium with bonds to group 15 elements such as $R_3Ga \cdot ER'_3$ ² ($E=N, P, As$) and $(R_2GaER'_2)_2$ ³ ($E=P, As$) have been receiving increasing attention due to their potential usefulness as precursors for the preparation of films of group 13-15 materials.⁴ In contrast, similar types of compounds with bonds to the heavier group 16 elements have been the subject of only limited research. The early work of G. E. Coates⁵ revealed that $GaMe_3$ reacted with EMe_2 ($E=Se, Te$) to form 1:1 adducts which were extensively dissociated in the vapor phase. Thus, the simple adduct $Me_3Ga \cdot TeMe_2$ is the only example of a reported compound containing a Ga-Te bond prior to the present work. When selenols⁶ were used, elimination reactions occurred at room temperature and dimeric derivatives of the type $(Me_2GaER)_2$ ($E=Se; R=Me, Ph$) were formed. These dimers were stable to dissociation in the gas phase at moderate temperatures but the gallium-chalcogen bond was cleaved by NMe_3 to give monomeric species of the type $Me_2GaER \cdot NMe_3$.⁶ The only other examples of derivatives containing Ga-Se bonds are $[Et_2GaSe(SiEt_3)]_2$,⁷ $[i-Pr(Br)GaSeEt]_3$,⁸ and $(EtGaSe)_n$.⁷

The first example of an organometallic gallium-tellurium compound $(Me_3CCH_2)_2GaTePh$, which is not a simple adduct, has been prepared by the reaction of $Ga(CH_2CMe_3)_2Cl$ with $LiTePh$ in Et_2O . The new compound has been fully characterized by partial elemental analyses (C and H), physical



properties, infrared spectroscopy, 1H and ^{125}Te NMR spectroscopies and an X-ray structural study. The tellurium reagent⁹ $LiTePh$ was generated by the reductive cleavage of Te_2Ph_2 by $LiBEt_3H$ in THF/Et_2O . The use of a slight excess of Te_2Ph_2 , which was readily removed by washing the $LiTePh$ with

pentane, insured that no LiEt₃H would be available to react with Ga(CH₂CMe₃)₂Cl in the second step of the synthesis. The reagent LiEt₃H has been observed previously to ethylate GeCl₄.¹⁰ The product (Me₃CCH₂)₂GaTePh was purified by repetitive low temperature (-20 °C) crystallizations from pentane and was isolated in low yield (20%) as an off-white, extremely air-sensitive solid. An unidentified orange, oily product, which was shown to contain tellurium by ¹²⁵Te NMR spectroscopy, prevented the isolation of the above product in higher yield.

The structure of (Me₃CCH₂)₂GaTePh consists of discrete dimeric units with the formula [(Me₃CCH₂)₂GaTePh]₂. There were no abnormally close contacts between dimeric units in the unit cell. The labelling of the atoms in the molecule is shown in Figure 1. The geometry of the Ga₂Te₂ butterfly ring is depicted in Figure 2. The interatomic distances and angles are collected in Tables I and II.

The Ga-Te bond distances in [(Me₃CCH₂)₂GaTePh]₂ are in the range of 2.7435(8) to 2.7623(8) Å. The average Ga-Te bond of 2.755 Å is 0.14 Å longer than the sum of the Ga and Te covalent radii, 2.62 Å.¹¹ Although no molecular compounds which contain Ga-Te bonds have been structurally characterized, Ga-Te bond distances have been reported for four GaTe based solid state phases. The average Ga-Te bond distances for the solid state phases GaTe (monoclinic),^{12,13} GaTe (hexagonal)¹⁴ and GaTeCl¹⁵ are 2.665 Å, 2.61 Å and 2.634 Å, respectively. The Ga₂Te₅ phase contains GaTe₄ tetrahedra with a Ga-Te bond distance of 2.641 Å³.

The four membered ring in [(Me₃CCH₂)₂GaTePh]₂ has a butterfly arrangement as found in [I₂GaS(i-Pr)]₂.¹⁶ The angle of fold about the gallium atoms in [(Me₃CCH₂)₂GaTePh]₂ is 150.21° and about the tellurium

atoms is 148.01° . The angle of fold about the gallium atoms in $[I_2GaS(i-Pr)]_2$ is $143.3(2)^\circ$.¹⁶ Interestingly, the ring systems in the dimers $[Ph_2GaSET]_2$ ^{17,18} and $[I_2GaSMe]_2$ ^{17,19} are planar. The phenyl groups in $[(Me_3CCH_2)_2GaTePh]_2$ are in the anti-position relative to the ring. The methyl groups in $[I_2GaSMe]_2$ ^{17,19} are also in the anti-position relative to the planar ring as are the ethyl groups in $[Ph_2GaSET]_2$.^{17,18} However, $[I_2GaS(i-Pr)]_2$, which has a butterfly ring geometry, has the i-propyl groups in the syn-position.¹⁶

Each gallium and tellurium atom in $[(Me_3CCH_2)_2GaTePh]_2$ has a distorted tetrahedral coordination environment. The Te-Ga-Te angles are 91.92° and 92.12° . These angles are similar to the S-Ga-S angles of 94.3° in $[I_2GaSMe]_2$, 93.6° in $[I_2GaS(i-Pr)]_2$ and 89.1° in $[Ph_2GaSET]_2$.¹⁷ The Ga-Te-Ga angles in $[(Me_3CCH_2)_2GaTePh]_2$ are 83.51° and 83.04° . These angles are similar to the Ga-S-Ga angles of 86.4° in $[Ph_2GaSET]_2$, 85.6° in $[I_2GaSMe]_2$ and 84.7° in $[I_2GaS(i-Pr)]_2$.¹⁶

The NMR spectral properties of the new gallium-tellurium compound are consistent with the presence of dimeric molecules $[(Me_3CCH_2)_2GaTePh]_2$ in solution. The ^{125}Te NMR spectrum, the most useful for identifying components, exhibits only one line at 345.4 ppm. For comparison, a solution of LiTePh in THF gave a line at 133.9 ppm whereas the unknown impurity which was an orange oil had a line at 370.1 ppm. The 1H NMR spectrum in benzene- d_6 had a broadened line at 1.30 ppm for the methylene protons of the neopentyl group and at least six lines of decreasing intensity from 1.11 to 0.81 ppm for the methyl protons of the neopentyl group. The phenyl lines were obscured by those due to the solvent. The occurrence of multiple and/or broadened lines for the neopentyl group protons suggests isomers but their presence could not be confirmed. Since the X-ray structural study

revealed only the trans isomer with the phenyl rings in the anti-position relative to the ring, the gallium-tellurium bonds in the dimer must have broken at least partially for isomerization to have occurred.

Experimental Section

All compounds described in this investigation were extremely sensitive to oxygen and moisture and were manipulated in a standard vacuum line or in a purified argon atmosphere. The alkyl gallium chloride, $\text{Ga}(\text{CH}_2\text{CMe}_3)_2\text{Cl}$ was prepared and purified by literature methods.^{20a} The reagent LiBEt_3H (1.0M in THF) was obtained from Aldrich Chemical Co. whereas Te_2Ph_2 was prepared as previously described.^{20b} Solvents were dried by conventional procedures. Elemental analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, NY. Infrared spectra of Nujol mulls between CsI plates were recorded by means of a Perkin-Elmer 683 spectrometer. Absorption intensities are reported with the abbreviations vs (very strong), s (strong), m (medium), w (weak) and sh (shoulder). The ^1H NMR spectra were recorded at 90 MHz by using a Varian EM-390 spectrometer. Proton chemical shifts are reported in δ units (ppm) and are referenced to SiMe_4 at δ 0.00 and benzene as δ 7.13 ppm. The ^{125}Te NMR spectra were recorded at 94.70 MHz by using a Varian XLAA-300 spectrometer. The proton decoupled ^{125}Te NMR spectra are reported relative to diphenyl telluride (TePh_2) as δ = 688 ppm, which was referenced to external dimethyl telluride (TeMe_2) as δ = 0.00 ppm. The samples were run unlocked and the chemical shifts were measured by tube interchange. All samples for NMR spectra were contained in sealed NMR tubes. Melting points were observed in sealed capillaries.

Synthesis of $(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}$.

In a glove box, a side-arm dumper was charged with 0.749 g of LiTePh (ca. 3 mmol) which had been prepared⁹ from Te_2Ph_2 and LiBEt₃H in THF/Et₂O and connected to a two-neck round-bottom flask charged with 0.871 g (3.52 mmol) of $\text{Ga}(\text{CH}_2\text{CMe}_3)_2\text{Cl}$. Diethyl ether (50 mL) was vacuum distilled into the reaction vessel and the contents were warmed to ambient temperature with stirring. The LiTePh was slowly added over a 5 min time period to the ether solution. The colorless solution slowly became yellow in color. The mixture was allowed to stir 16 h and the Et₂O was removed by vacuum distillation. An orange colored oil remained in the flask. The vessel was fitted with a medium frit and a Schlenk receiving flask in the glove box. Anhydrous pentane (20 mL) was distilled into the flask and repetitive low temperature crystallizations (-10 to -20 °C) provided 0.336 g (0.807 mmol, 22.9% yield) of an off-white solid which was identified as $(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}$. Recrystallization of $(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}$ from pentane at -20 °C afforded crystallographic quality crystals.

$(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}$. mp 109.0-110.5 °C dec (sample begins to decompose and darken at 96 °C and melts to an orange liquid at 109.0-110.5 °C). ¹H NMR (C_6D_6 , δ): 0.81, 0.89, 0.96, 1.01, 1.05, 1.11 (s, combined lines 18 H, -CMe₃); 1.30 (s, 3.7 H, -CH₂-). ¹²⁵Te{¹H} (C_6D_6 , δ): 345.4 (s). The ¹²⁵Te NMR spectrum of the orange oil from the reaction gave a resonance in the ¹²⁵Te{¹H} NMR spectrum at 370.1 ppm. The ¹²⁵Te NMR spectrum of a sample of LiTePh in THF gave a resonance in the ¹²⁵Te{¹H} NMR spectrum at 133.9 ppm. Anal. Calcd. for $\text{C}_{32}\text{H}_{54}\text{Ga}_2\text{Te}_2$: C, 46.12; H, 6.53. Found: C, 46.41, H, 6.63. IR (Nujol, cm^{-1}): 3070 (vw), 3055 (vw), 1571 (m), 1430 (m), 1358 (m), 1232 (m), 1133 (m), 1099 (m), 1062 (vw), 1055 (vw), 1011 (m), 999 (w,sh), 994 (m), 795 (vw,br), 740 (w), 720 (s), 648 (w), 618 (m), 449 (w).

Crystallography. A colorless crystal of $(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}$ was selected and sealed in a glass capillary under an argon atmosphere for examination by X-ray diffraction procedures. Data were collected on an Enraf-Nonius CAD-4 diffractometer by using graphite monochromated Mo K α radiation. Table III summarizes the unit cell data and the refinement parameters. Unit cell dimensions were determined by least-squares refinement of 25 reflections ($20.0^\circ < 2\theta < 26.8^\circ$). Three standard reflections were remeasured at every hour of X-ray exposure. The data were corrected for a slow, steady decrease of intensity over time. The maximum correction was 1.230 on I. An empirical absorption correction²¹ was applied. Correction factors ranged from 0.85 to 1.31.

The structure of $(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}$ was solved by direct methods by using MULTAN 11/82.²² An E-map phased with the best phase set gave the four heavy atom positions. Subsequent difference electron density maps allowed location of the remaining atoms. All hydrogen atoms were included in the refinement except that those on methyl groups were fixed at calculated positions riding on the parent carbon atoms. Positional parameters, excluding hydrogen atoms, are given in Table IV.

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Supplementary Material Available. Table of refined temperature factors for $[(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}]_2$ (2 pages); table of hydrogen atom positional parameters (3 pages); tables of carbon-carbon bond distances and angles (2 pages); structure factors (29 pages). Ordering information is given on any current masthead page.

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Table I. Interatomic Distances (\AA) for $[(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}]_2$.

(A) Gallium-Tellurium Distances

Ga(1)-Te(1)	2.7623(8)	Ga(2)-Te(2)	2.7525(8)
Ga(1)-Te(2)	2.7435(8)	Ga(2)-Te(1)	2.7623(8)

(B) Gallium-Carbon Distances

Ga(1)-C(7)	1.975(7)	Ga(2)-C(23)	1.988(7)
Ga(1)-C(12)	1.962(8)	Ga(2)-C(28)	1.979(8)

(C) Tellurium-Carbon Distances

Te(1)-C(1)	2.125(6)	Te(2)-C(17)	2.125(6)
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Table II. Angles (in deg) for $[(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}]_2$.

(A) Angles around the Gallium Atom

Te(1)-Ga(1)-Te(2)	92.1(2)	Te(1)-Ga(2)-Te(2)	91.92(2)
Te(1)-Ga(1)-C(7)	103.5(2)	Te(1)-Ga(2)-C(23)	104.4(2)
Te(1)-Ga(1)-C(12)	111.6(4)	Te(2)-Ga(2)-C(28)	114.2(3)
Te(2)-Ga(1)-C(7)	108.9(3)	Te(1)-Ga(2)-C(23)	103.7(3)
Te(2)-Ga(1)-C(12)	103.7(3)	Te(1)-Ga(2)-C(28)	105.8(2)
C(7)-Ga(1)-C(12)	130.4(4)	C(23)-Ga(2)-C(28)	129.7(4)

(B) Angles around the Tellurium Atom

Ga(1)-Te(1)-Ga(2)	83.51(2)	Ga(1)-Te(2)-Ga(2)	84.04(2)
Ga(1)-Te(1)-C(1)	102.6(2)	Ga(1)-Te(2)-C(17)	99.1(2)
Ga(2)-Te(1)-C(1)	106.8(2)	Ga(2)-Te(2)-C(17)	103.8(2)

(C) Gallium-Carbon-Carbon Angles

Ga(1)-C(7)-C(8)	121.6(5)	Ga(2)-C(23)-C(24)	121.3(6)
Ga(1)-C(12)-C(13)	123.1(6)	Ga(2)-C(28)-C(29)	122.7(6)

(D) Tellurium-Carbon-Carbon Angles

Te(1)-C(1)-C(2)	121.1(5)	Te(2)-C(17)-C(18)	122.7(5)
Te(1)-C(1)-C(6)	119.4(5)	Te(2)-C(17)-C(21)	118.2(6)

Table III. Summary of Crystal Data and Refinement Parameters

Formula	$(\text{GaTeC}_{16}\text{H}_{27})_2$
M.W.	833.428
Crystal system	Monoclinic
Space group	P2 ₁ /C
Cell constants at 23(1)°C: a, Å	18.864(7)
b	10.041(5)
c	19.869(9)
B, deg	101.80(3)
V, Å ³	3684(5)
No. molecules/unit cell (Z)	4
D(calc), g cm ⁻³	1.505
Crystal dimensions, mm	0.18 x 0.25 x 0.38
Absorption coeff. (μ , Mo Ka) cm ⁻¹	30.4
Scan technique	ω -2θ
Scan rate, deg 2θ min ⁻¹	2.1 to 20
2θ limit, deg	46
hkl range	-20 to +20, 0 to 11, 0 to 21
No. of unique data measured	5100
No. of data used in refinement ($I > o(I)$)	3954
No. of parameters	397
$R = \sum F_o - K F_c / \sum F_o $	0.030
$R_w = (\sum w(F_o - K F_c)^2 / \sum w F_o ^2)^{1/2}$	0.035
$S = [(\sum w(F_o - K F_c)^2 / (n_o - n_v))]^{1/2}$	0.94
Wtg. parameters ($w^{-1} = o^2(F_o) + (pF_o)^2 + q$): P	0.02
	q 1.0
Scale factor, K	0.6497(2)
Maximum shift in final cycle, (Δ/o)	0.03
Residual electron density in final difference Fourier synthesis (e/Å ³)	-0.33 to +0.44

Table IV. Positional Parameters (Esd's in Parentheses) for
 $[(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}]_2^{\text{a}}$.

<u>Atom</u>	<u>x</u>	<u>y</u>	<u>z</u>	<u>B(Å²)</u>
TE1	0.23526(2)	0.46017(4)	0.36194(2)	4.466(8)
TE2	0.19798(2)	0.76885(4)	0.23521(2)	4.453(8)
GA1	0.21570(3)	0.73101(6)	0.37440(3)	4.23(1)
GA2	0.27140(3)	0.53118(6)	0.23890(3)	4.51(1)
C1	0.3341(3)	0.4253(5)	0.4331(3)	4.5(1)
C2	0.3915(4)	0.3614(6)	0.4131(3)	5.9(2)
C3	0.4536(4)	0.3314(7)	0.4628(4)	6.9(2)
C4	0.4585(4)	0.3626(7)	0.5299(4)	7.5(2)
C5	0.4015(4)	0.4245(8)	0.5486(4)	7.6(2)
C6	0.3391(4)	0.4567(6)	0.5012(3)	6.0(2)
C7	0.3127(3)	0.7953(6)	0.4196(3)	5.2(1)
C8	0.3333(3)	0.9419(6)	0.4185(3)	5.2(1)
C9	0.2822(5)	1.0242(8)	0.4459(7)	17.2(4)
C10	0.4083(5)	0.9634(8)	0.4601(5)	11.5(3)
C11	0.3362(6)	0.9864(9)	0.3475(5)	15.9(3)
C12	0.1199(3)	0.7718(8)	0.3933(3)	6.8(2)
C13	0.0990(3)	0.7417(6)	0.4618(3)	5.7(2)
C14	0.0880(7)	0.604(1)	0.4688(6)	25.1(4)
C15	0.0313(5)	0.807(2)	0.4689(5)	20.1(5)
C16	0.1528(6)	0.783(2)	0.5171(5)	24.7(6)
C17	0.0874(3)	0.7130(6)	0.2050(3)	4.8(1)
C18	0.0615(3)	0.5929(6)	0.2226(4)	6.1(2)
C19	-0.0115(4)	0.5641(8)	0.2035(4)	7.9(2)

C20	-0.0573(4)	0.6533(9)	0.1660(4)	8.7(2)
C21	-0.0322(4)	0.7726(8)	0.1474(5)	8.4(2)
C22	0.0401(3)	0.8023(7)	0.1673(4)	6.3(2)
C23	0.3749(3)	0.5842(7)	0.2647(3)	5.9(2)
C24	0.4163(3)	0.6338(7)	0.2106(4)	6.6(2)
C25	0.4936(4)	0.669(1)	0.2451(5)	12.3(3)
C26	0.4235(5)	0.517(1)	0.1624(5)	11.2(3)
C27	0.3777(5)	0.7415(9)	0.1699(5)	15.5(3)
C28	0.2202(4)	0.4281(6)	0.1586(3)	5.8(2)
C29	0.2182(4)	0.2763(6)	0.1577(3)	6.2(2)
C30	0.2924(5)	0.2199(8)	0.1877(5)	9.9(3)
C31	0.1643(5)	0.2253(8)	0.1994(4)	9.5(2)
C32	0.1925(5)	0.2272(8)	0.0834(4)	9.4(2)

^aAnisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as (4/3) [a²B(1,1) + b²B(2,2) + c²B(3,3) + ab(cos Y)B(1,2) + ac(cos B)B(1,3) + bc(cos a)B(2,3)].

Captions for Figures

Figure 1. ORTEP Plot of $[(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}]_2$. The thermal ellipsoids were drawn at the 40% probability level.

Figure 2. Geometry of Ga_2Te_2 Ring in $[(\text{Me}_3\text{CCH}_2)_2\text{GaTePh}]_2$.

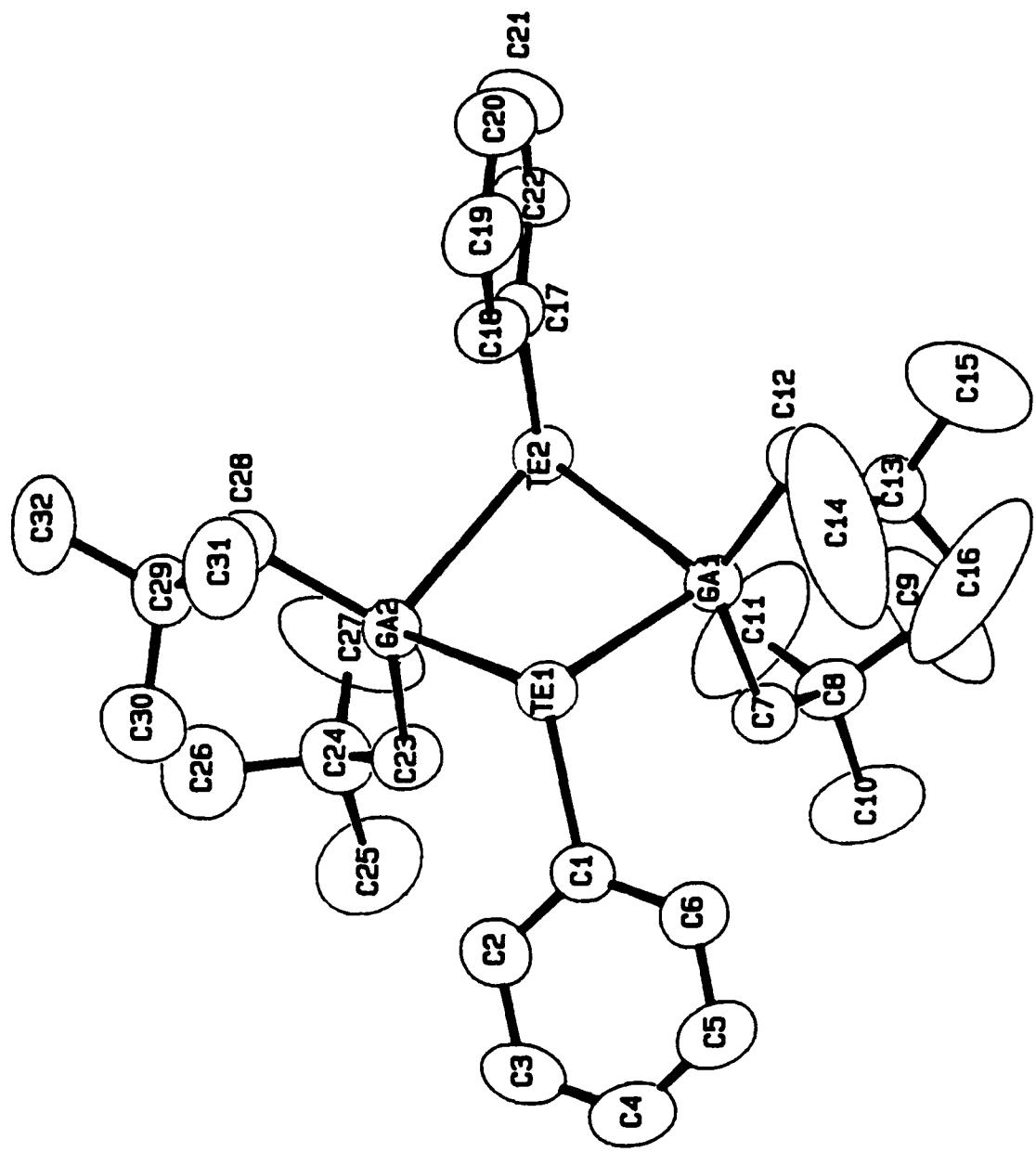


Fig 1

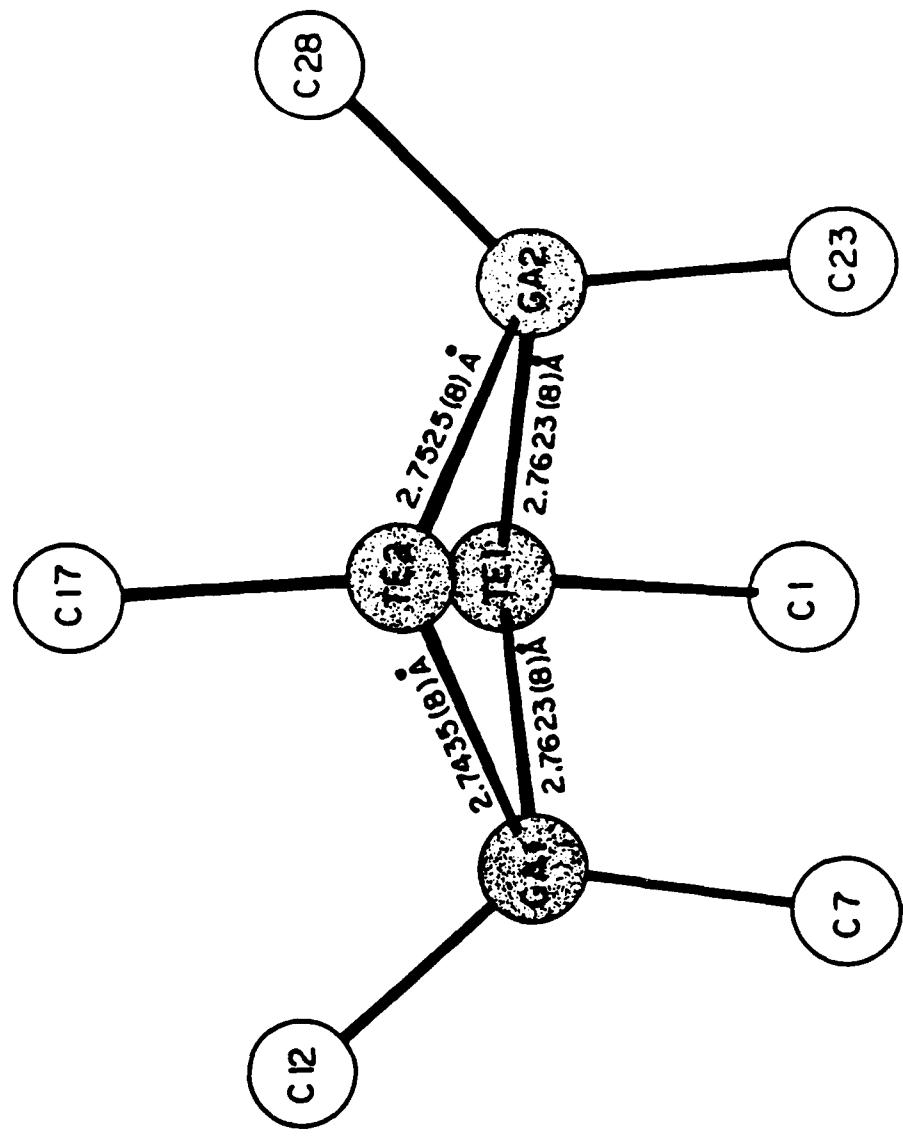


Fig 2